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(71)Applicant : NATIONAL INSTITUTE OF ADVANCED
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(54) METHOD FOR PRODUCING SILICA RAW MATERIAL

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a new method for producing, from rice chaff, a silica raw material which is suitable as a raw material for producing various silicon compounds, especially silicon containing ceramics.

SOLUTION: The silica raw material is produced by treating rice chaff powder with compressed hot water of 150 to 450°C for at least 30 s. The contents of a silica component and an organic component contained in the produced silica raw material are controlled by controlling the reaction temperature and the reaction time.

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METHOD FOR PRODUCING SILICA RAW MATERIAL

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Inventor: SAKAKI TAKESHI; KOMATSU MASAHIRO; KUMAGAI SATOSHI; HAYASHI NOBUYUKI

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Abstract of JP2002265257

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(54)【発明の名称】シリカ原料の製造方法

(57)【要約】

【課題】 各種ケイ素化合物、特に含ケイ素セラミック
の製造用原料として適したシリカ原料をもみ殻から製
造するための新規な方法を提供する。

【解決手段】 もみ殻粉末を150～450℃の加圧熱
水で少なくとも30秒間処理し、反応温度及び反応時間
を制御することにより、生成するシリカ原料中のシリカ
成分と有機質分の含有割合を調整してシリカ原料を製造
する。

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【特許請求の範囲】

【請求項1】 もみ殻粉末を150～450℃の加圧熱水で少なくとも30秒間処理することを特徴とするシリカ原料の製造方法。

【請求項2】 反応温度及び反応時間を制御することにより、生成するシリカ原料中のシリカ成分と有機質分の含有割合を調整する請求項1記載のシリカ原料の製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、各種ケイ素化合物、例えば含ケイ素セラミックスの製造用原料として好適なシリカ原料をもみ殻から製造するための新規な方法に関するものである。

【0002】

【従来の技術】農産廃棄物であるもみ殻中には、リグニン、ヘミセルロース、セルロースなどの有機質分と、17～22質量%程度の大部分がアモルファスシリカからなる無機質分（灰分）が含まれ、さらにカリウムのようなアルカリ金属類が微量含まれていることが知られている。

【0003】このため、有機質の炭素によるSiO₂の熱還元反応を応用して、炭化ケイ素系セラミックスや塗化ケイ素系セラミックスを製造することが試みられているが、もみ殻をそのまま炭化すると、シリカに対する有機炭素の量が多いため、SiC生成物中に過剰の炭素が残留することになるし、また有機質分の一部は、炭化時に揮発して、製品を多孔質化するなどの問題を生じる。したがって、もみ殻からシリカ原料を製造する場合には、原料中の有機質分とシリカの含有割合を調整することが極めて重要になる。

【0004】ところで、これまで、もみ殻中のシリカ成分を抽出する方法としては、硝酸や微生物、あるいは蒸着・爆砕法により有機質分を分解する方法や、もみ殻を粉碎する際に、シリカ成分の割合の多い外皮部分が先に微粒化されることを利用した分級分離法（特公平7-12446号公報）などが知られている。

【0005】しかしながら、前記硝酸分解法においては、廃液処理の問題があるし、微生物法では処理時間が長すぎて専用的でない。一方、蒸着・爆砕法では操作が

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【課題を解決するための手段】本発明者らは、もみ殻からシリカ原料を製造する方法について種々研究を重ねた結果、反応温度と反応時間を制御することにより、もみ殻中のシリカと有機質分との含有割合を適正な範囲に調整しうること、したがってこれを利用すれば使用目的に応じた品質のシリカ原料を製造しうることを見出し、この知見に基づいて本発明を完成するに至った。

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【0006】すなわち、本発明は、もみ殻粉末を150～450℃の加圧熱水で少なくとも30秒間処理することを特徴とするシリカ原料の製造方法、及び反応温度及び反応時間を制御することにより、生成するシリカ原料中のシリカ成分と有機質分の含有割合を調整する前記記載のシリカ原料の製造方法を提供するものである。

【0009】

【発明の実施の形態】本発明方法においては、原料としてもみ殻を粉碎処理して得られたもみ殻粉末が用いられる。このもみ殻粉末の粒度については特に制限はないが、32メッシュ篩目を通過する程度の粒度を有するものが好ましい。

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【0010】本発明方法においては、上記もみ殻粉末を150～450℃の加圧熱水と接触させ、もみ殻中の有機質分を抽出除去すると共に、分解させて、もみ殻中のシリカ成分と有機質分との含有割合を調整する。このシリカ成分と有機質分との含有割合は、シリカの用途に応じて選定され、例えば炭化ケイ素（SiC）系セラミックスを製造する場合には、もみ殻中の有機質分の炭素原子とシリカ成分のケイ素原子との比が、化学量論的比の近傍になるように調整するのが有利である。この加圧熱水との接触に際しては、不活性ガス雰囲気、例えば窒素やアルゴン中で行うのが好ましい。

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【0011】上記加圧熱水の温度が150℃未満では、もみ殻中の有機質分の抽出除去及び分解の速度が遅すぎて、もみ殻中のシリカ成分と有機質分との含有割合が所望の値になるまでに長時間を要し、実用的でない。一方、加圧熱水が450℃を超えると系の圧力が高くなりすぎ、それに耐える高圧装置を必要とし、設備費などを考慮すると、この加圧熱水の好ましい温度は200～400℃の範囲であり、特に250～350℃の範囲が好適である。

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させることにより、もみ殻中の有機質分は、一部水可溶分となって抽出除去されると共に、一部分解してガス化される。上記水可溶分は、250°C近傍の加圧熱水を用いる場合は、キシロース及びキシロオリゴ糖が主体であり、またそれより高い温度の加圧熱水を用いる場合は、グルコース及びセロオリゴ糖が主体となる。したがって、この水可溶分は、食品添加剤として有用なオリゴ糖として利用可能である。

【0014】この加圧熱水による接触処理において、例えば灰分17質量%、灰分中のS：8.9質量%のもみ殻粉末を用いた場合、温度250～350°C、時間0～90秒の処理条件で、水不溶分中の灰分含有量は、17～3.9質量%の範囲に濃縮される。したがって、加圧熱水の温度及び処理時間を適当に選択することにより、灰分含有量を17～3.9質量%の範囲で、シリカ成分と有機質分との含有割合を所望の値に調整することができる。

【0015】本発明方法によれば、灰分中の全金属成分量に基づくS：濃度は、未処理もみ殻灰分中のS：濃度が約8.9質量%であるのに対し、9.6. 5～9.9. 5質量%という高純度シリカを含む灰分が得られる。これは灰分中のアルカリ金属類が加圧熱水処理により除去されるためである。

【0016】

【実施例】次に実施例により、本発明をさらに詳細に説明するが、本発明は、これらの例によってなんら限定されるものではない。

【0017】実施例1

3.2 メッシュ以下に粉碎し、乾燥したもみ殻粉末（灰分17.4質量%、灰分中の全金属成分量に基づくS：濃度8.9. 1質量%）0.5gと蒸留水3.0gを内容積5.6mlのバルブ付ステンレス製鋼反応容器に仕込み、空気を窒素ガスで置換したのち、バルブを閉じて系内を密閉した。この反応器を予め所定温度に加熱しておいた塩浴中に投入し、所定時間振とうして反応させたのち、水浴中にて急冷して反応を終結させた。反応器内のガスを排出したのち、内容物をG4ガラスフィルター上に取り出し、ろ過してろ液と残さを採取し、それぞれ減圧蒸留及び真空乾燥して水分を除去し、水可溶分（WS）と水不溶分（WI）を得た。それぞれの仕込み原料基準の収率（質量%）を求め、また質量損失率からガス

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1）中のシリカを含む灰分は17.4質量%から21.3質量%まで濃縮された。

【0018】実施例2

反応温度が300°Cになるように塩浴温度を設定し、それ以外は実施例1と同じ条件で反応を行った際の反応生成物収率及び水不溶分（WI）中のシリカを含む灰分の濃度と反応時間の関係を図2に示す。図2から分るよう、各生成物の収率は実施例1と同様の変化を示したが、反応時間90秒で水不溶分（WI）収率は5.7質量%にまで減少し、それに伴い、水不溶分（WI）中の灰分は17質量%から2.9質量%にまで濃縮できた。

【0019】実施例3

反応温度が350°Cになるように塩浴温度を設定し、それ以外は実施例1と同じ条件で反応を行った際の反応生成物の収率及び水不溶分（WI）中のシリカを含む灰分の濃度と反応時間の関係を図3に示す。図3から分るよう、各生成物の収率は実施例1及び2と同様の変化を示したが、反応時間90秒で水不溶分（WI）収率は4.1質量%にまで減少し、それに伴い、そのシリカを含む灰分の濃度は3.9質量%にまで濃縮できた。また水不溶分（WI）中のシリカを含む灰分の濃度と反応温度の関係を調べるため、実施例1～3のデータ中で、反応時間を30秒あるいは90秒とした際の水不溶分（WI）中のシリカを含む灰分の濃度と反応温度の関係を図4にプロットした。図4から分るよう、シリカを含む灰分の濃度は反応温度及び反応時間の上昇と共に増加しており、反応温度及び反応時間を250～350°C、0～90秒の間で適当に選定することにより、水不溶分（WI）中のシリカを含む灰分の濃度を17～3.9質量%の間で任意に制御できることが確認できた。

【0020】次に、反応時間30秒の処理で得られた水不溶分中の灰分の元素分析をエネルギー分散型ケイ光X線分析装置を用いて行った結果、未処理もみ殻中の灰分の全金属成分量に基づくS：濃度が8.9. 1質量%に対し、250°C処理のそれは9.6. 7質量%、300°C処理のそれは9.7. 6質量%、そして350°C処理のそれは9.9. 3質量%にまで向上しており、本法で濃縮された無機質分は極めて純度の高いシリカから成ることが分った。

【0021】さらに、本処理によって得られる水可溶分

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易に制御することができ、使用目的に応じた高品質のシリカ原料、例えば炭化ケイ素系セラミックス製造用のシリカ原料を製造することができる。

【図面の簡単な説明】

【図1】 反応温度250°Cの場合の各反応生成物の収率及び水不溶分(WI)中の灰分濃度と反応時間の関係を示すグラフ。

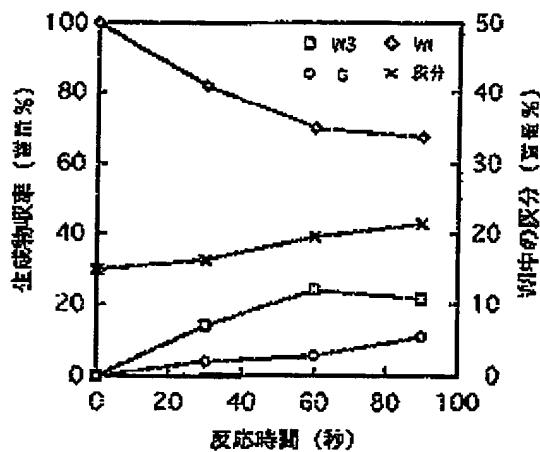
【図2】 反応温度300°Cの場合の各反応生成物の収率及び水不溶分(WI)中の灰分濃度と反応時間の関係を示すグラフ。

*率及び水不溶分(WI)中の灰分濃度と反応時間の関係を示すグラフ。

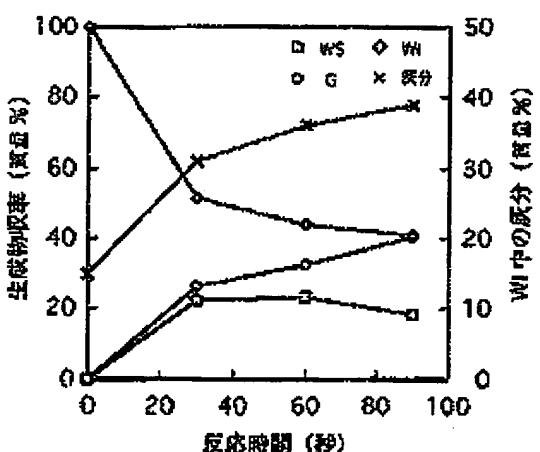
【図3】 反応温度350°Cの場合の各反応生成物の収率及び水不溶分(WI)中の灰分濃度と反応時間の関係を示すグラフ。

【図4】 反応時間30秒若しくは90秒とした場合の水不溶分(WI)中の灰分濃度と反応温度の関係を示すグラフ。

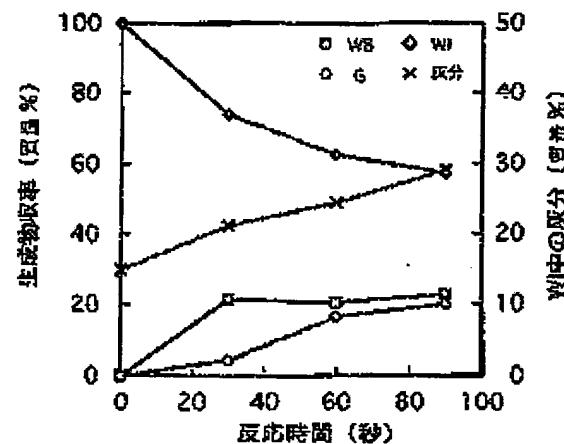
【図1】



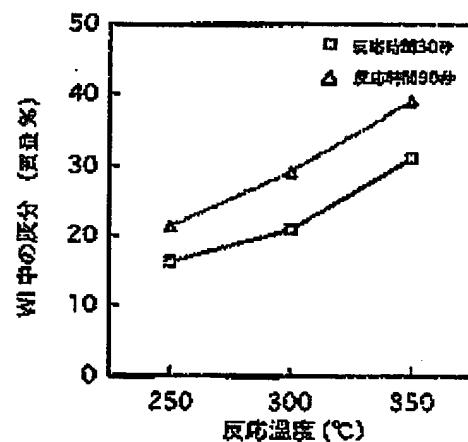
【図3】



【図2】



【図4】



English Translation of JP2002265257
Computer Translation from Japanese Patent Office Web Site
June 21, 2004

[Field of the Invention] This invention relates to the new approach for manufacturing a silica raw material suitable as a raw material for manufacture of various silicon compounds, for example, the ** silicon ceramics, from chaff.

[Description of the Prior Art] in the chaff which is agricultural production trash, the amount of [which organic parts, such as a lignin, a hemicellulose, and a cellulose, and most 17 - 22 mass % extent become from an amorphous silica] (ash content) minerals contain -- having -- alkaline metals still like a potassium -- ***** rare ***** -- things are known.

[0003] For this reason, since there are many amounts of the organic carbon to a silica when chaff is carbonize as it is although to apply the heat reduction reaction of SiO₂ by the carbon of the quality of organic, and to manufacture the silicon carbide system ceramics and the silicon nitride system ceramics is try, superfluous carbon will remain in a SiC product, and a part of organic part volatilizes at the time of carbonization, and it produces the problem of porosity-ize a product. Therefore, in manufacturing a silica raw material from chaff, it becomes very important to adjust the content rate of the organic part in a raw material and a silica.

[0004] By the way, as an approach of condensing the silica component in chaff, the approach of decomposing an organic part by the nitric acid, the microorganism, or *** and the exploding method, the classification separation method (JP,7-12446,B) which used that an envelope part with many rates of a silica component was atomized previously when pulverizing chaff are known until now.

[0005] However, in said nitric-acid part solution method, there is a problem of waste fluid processing, and by the microorganism method, the processing time is too long and is not practical. On the other hand, in order for there to be a fault with the silica unrecoverable [in / there are many energy losses the top where actuation is complicated, and / a classification separation method] by *** and the exploding method in coarse grain and to have adjusted the silica component in chaff, it was not necessarily a suitable approach.

[Brief Description of the Drawings]

[Drawing 1] The graph which shows the yield of each resultant in the case of 250-degree C reaction temperature, and the ash content concentration in a water-insoluble part (WI) and the relation of reaction time.

[Drawing 2] The graph which shows the yield of each resultant in the case of 300-degree C reaction temperature, and the ash content concentration in a water-insoluble part (WI) and the relation of reaction time.

[Drawing 3] The graph which shows the yield of each resultant in the case of 350-degree C reaction temperature, and the ash content concentration in a water-insoluble part (WI) and the relation of reaction time.

[Drawing 4] The graph which shows the relation of the ash content concentration and reaction temperature in the water-insoluble part (WI) at the time of considering as reaction-time 30 seconds or, and 90 seconds.

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the new approach for manufacturing a silica raw material suitable as a raw material for manufacture of various silicon compounds, for example, the ** silicon ceramics, from chaff.

[0002]

[Description of the Prior Art] in the chaff which is agricultural production trash, the amount of [which organic parts, such as a lignin, a hemicellulose, and a cellulose, and most 17 - 22 mass % extent become from an amorphous silica] (ash content) minerals contain -- having -- alkaline metals still like a potassium -- ***** rare ***** -- things are known.

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[0004] By the way, as an approach of condensing the silica component in chaff, the approach of decomposing an organic part by the nitric acid, the microorganism, or **** and the exploding method, the classification separation method (JP,7-12446,B) which used that an envelope part with many rates of a silica component was atomized previously when pulverizing chaff are known until now.

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[0006]

[Problem(s) to be Solved by the Invention] This invention is made for the purpose of offering the new approach for manufacturing from chaff the silica raw material which was suitable as a raw material for manufacture of various silicon compounds, especially the ** silicon ceramics.



[0007]

[Means for Solving the Problem] As a result of repeating research variously about the approach of manufacturing a silica raw material from chaff, this invention persons came to complete this invention for the ability of the silica raw material of quality according to the purpose of use to be manufactured by controlling reaction temperature and reaction time based on a header and this knowledge, when using that the content rate of the silica in chaff and an organic part can be adjusted to the proper range, therefore this.

[0008] That is, this invention offers the manufacture approach of the silica raw material said publication of adjusting the content rate of the silica component in the silica raw material to generate, and an organic part, by controlling the manufacture approach, reaction temperature, and reaction time of the silica raw material characterized by processing chaff powder for at least 30 seconds with 150-450-degree C pressurization hot water.

[0009]

[Embodiment of the Invention] In this invention approach, the chaff powder obtained by carrying out grinding processing of the chaff as a raw material is used. Although there is especially no limit about the grain size of this chaff powder, what has the grain size of extent which passes a 32-mesh mesh is desirable.

[0010] In this invention approach, while contacting the above-mentioned chaff powder to 150-450-degree C pressurization hot water and carrying out extract removal of the organic part in chaff, it is made to decompose and the content rate of the silica component in chaff and an organic part is adjusted. When it is selected according to the application of a silica, for example, manufactures the silicon carbide (SiC) system ceramics, as for the content rate of this silica component and an organic part, it is advantageous that the ratio of the carbon atom of the organic part in chaff and the silicon atom of a silica component adjusts so that it may become near the stoichiometric ratio. It is desirable to carry out in an inert gas ambient atmosphere, for example, nitrogen, and an argon on the occasion of contact to this pressurization hot water.

[0011] The temperature of the above-mentioned pressurization hot water takes long duration for the rate of extract removal of the organic part in chaff and decomposition to be too slow, and for the content rate of the silica component in chaff and an organic part to become a desired value at less than 150 degrees C, and is not practical. On the other hand, if pressurization hot water exceeds 450 degrees C, it becomes high too much, and a system pressure will need the high pressure installation which bears it, and will become disadvantageous economically in respect of an installation cost. If a concentration rate, an installation cost, etc. of a silica component are taken into consideration, the range of the desirable temperature of this pressurization hot water is 200-400 degrees C, and the range of 250-350 degrees C is especially suitable for it.

[0012] Moreover, although the contact time of chaff powder and pressurization hot water is influenced by the content rate of a request of a silica component, the temperature of pressurization hot water, etc., it is usually a 30 - 120-second about room for at least 30 seconds. Furthermore, although there is especially no limit about the quantitative rate of chaff powder and pressurization hot water, when the concentration effectiveness of a silica component, the volume efficiency of equipment, etc. are taken into consideration, it is desirable to use [of 3 - 15 mass

section] pressurization hot water at a rate of 4 - 10 mass section comparatively especially to the chaff powder 1 mass section.

[0013] Thus, a part of organic part in chaff turns into water extractives by contacting chaff powder to pressurization hot water, and while extract removal is carried out, a part is understood and it is gasified. When about 250-degree C pressurization hot water is used for the above-mentioned water extractives, a xylose and a xylo oligosaccharide are subjects, and when using the pressurization hot water of temperature higher than it, a glucose and a celo oligosaccharide serve as a subject. Therefore, these water extractives are available as an oligosaccharide useful as a food additive.

[0014] In the contact processing by this pressurization hot water, when the chaff powder of ash content 17 mass % and Si89 mass % in ash content is used, the ash content in a water-insoluble part is condensed by the range of 17 - 39 mass % on the temperature of 250-350 degrees C, and the processing conditions for time amount 0 - 90 seconds. Therefore, ash content can be adjusted to the value of a request of the content rate of a silica component and an organic part in the range of 17 - 39 mass % by choosing suitably the temperature and the processing time of pressurization hot water.

[0015] As for Si concentration based on the total amount of metal components in ash content, according to this invention approach, the ash content in which Si concentration in unsettled chaff ash content contains the high grade silica 96.5 to 99.5 mass %, to being about 89 mass % is obtained. This is because the alkaline metals in ash content are removed by pressurization hot water processing.

[0016]

[Example] Next, although an example explains this invention to a detail further, this invention is not limited at all by these examples.

[0017] After grinding in the 132 or less meshes of examples, teaching dry 0.5g (ash content 17.4 mass %, Si concentration 89.1 mass % based on the total amount of metal components in ash content) of chaff powder and 3.0g of distilled water to the stainless steel steel-manufacture reaction container with a bulb of 5.6ml of content volume and nitrogen gas's permuting air, the bulb was closed and the inside of a system was sealed. After having thrown in this reactor in the salt bath beforehand heated to predetermined temperature, carrying out predetermined time shake and making it react, it quenched in the water bath and the reaction was made to end. take out and filter contents on G4 glass filter after discharging the gas in a reactor -- liquid and a residue -- extracting -- respectively -- vacuum distillation -- and the vacuum drying was carried out, moisture was removed and water extractives (WS) and a water-insoluble part (WI) were obtained. It asked for the yield (mass %) of each preparation raw material criteria, and gas and volatile-matter yield (G) were calculated from the amount of mass loss. Salt bath temperature is set up so that reaction temperature may become 250 degrees C, and the predetermined yield of each resultant at the time of carrying out a time amount reaction and the ash content concentration in a water-insoluble part (WI), and the relation of reaction time are shown in drawing 1 in this. Water solubilization of the chaff was carried out with the reaction passage of time, and water extractives (WS) yield reached about 25 mass % in 60 seconds so that drawing 1 might show. However, after it, gasification progressed and the inclination gas and whose

volatile-matter yield (G) increase was shown. The yield of chaff [a water-insoluble part (WI)] continued reduction with water solubilization, it became 67 mass % after 90 seconds, and the ash content containing the silica in a water-insoluble part (WI) was condensed from 17.4 mass % to 21.3 mass % in connection with it.

[0018] Salt bath temperature is set up so that example 2 reaction temperature may become 300 degrees C, and the concentration of ash content and the relation of reaction time containing the resultant yield at the time of reacting on the same conditions as an example 1 and the silica in a water-insoluble part (WI) are shown in drawing 2 except it. Although the yield of each product showed the same change as an example 1 so that drawing 2 might show, water-insoluble part (WI) yield decreased even to 57 mass % in reaction-time 90 seconds, and the ash content in a water-insoluble part (WI) has been condensed even from 17 mass % to 29 mass % in connection with it.

[0019] Salt bath temperature is set up so that example 3 reaction temperature may become 350 degrees C, and the concentration of ash content and the relation of reaction time containing the yield of the resultant at the time of reacting on the same conditions as an example 1 and the silica in a water-insoluble part (WI) are shown in drawing 3 except it. Although the yield of each product showed the same change as examples 1 and 2 so that drawing 3 might show, water-insoluble part (WI) yield decreased even to 41 mass % in reaction-time 90 seconds, and the concentration of the ash content containing the silica has been condensed even to 39 mass % in connection with it. Moreover, in order to investigate the relation containing the silica in a water-insoluble part (WI) of the concentration and reaction temperature of ash content, the relation of the concentration and reaction temperature of ash content which contains the silica in the water-insoluble part at the time of making reaction time into 30 seconds or 90 seconds (WI) in the data of examples 1-3 was plotted to drawing 4 . It has checked that the concentration of the ash content containing a silica could control to arbitration the concentration of the ash content which contains the silica in a water-insoluble part (WI) by increasing with the rise of reaction temperature and reaction time, and selecting reaction temperature and reaction time suitably in 250-350 degrees C and 0 - 90 seconds between 17 - 39 mass %s so that drawing 4 might show.

[0020] Next, the result of having performed elemental analysis of the ash content in a water-insoluble part to have been obtained by the processing for reaction-time 30 seconds using the energy dispersion mold cay light X-rays spectroscopic analyzer, Si concentration based on the total amount of metal components of the ash content in unsettled chaff receives 89.1 mass %. It turned out that the amount of [whose it of 250 degree-C processing is improving even to 99.3 mass % / by which it of 96.7 mass % and 300 degree-C processing was condensed / it / for it of 97.6 mass % and 350 degree-C processing by this method] minerals consist of a silica with very high purity.

[0021] Furthermore, about the water extractives (WS) obtained by this processing, as a result of the ion chromatography for sugar analysis analyzing the presentation, as for the water extractives from which a xylose and a xylo oligosaccharide are subjects, and, as for the water extractives obtained near 250 degree C, are obtained at the temperature beyond it, it turned out that a glucose and a cello oligosaccharide are subjects. That is, the water extractives obtained by this processing are available as an oligosaccharide useful as a food additive.

[0022]

[Effect of the Invention] According to this invention approach, it can be efficient in a short time, the content rate of the silica component in chaff and an organic part can be controlled easily, and the silica raw material of high quality according to the purpose of use, for example, the silica raw material for silicon carbide system ceramic manufacture, can be manufactured.

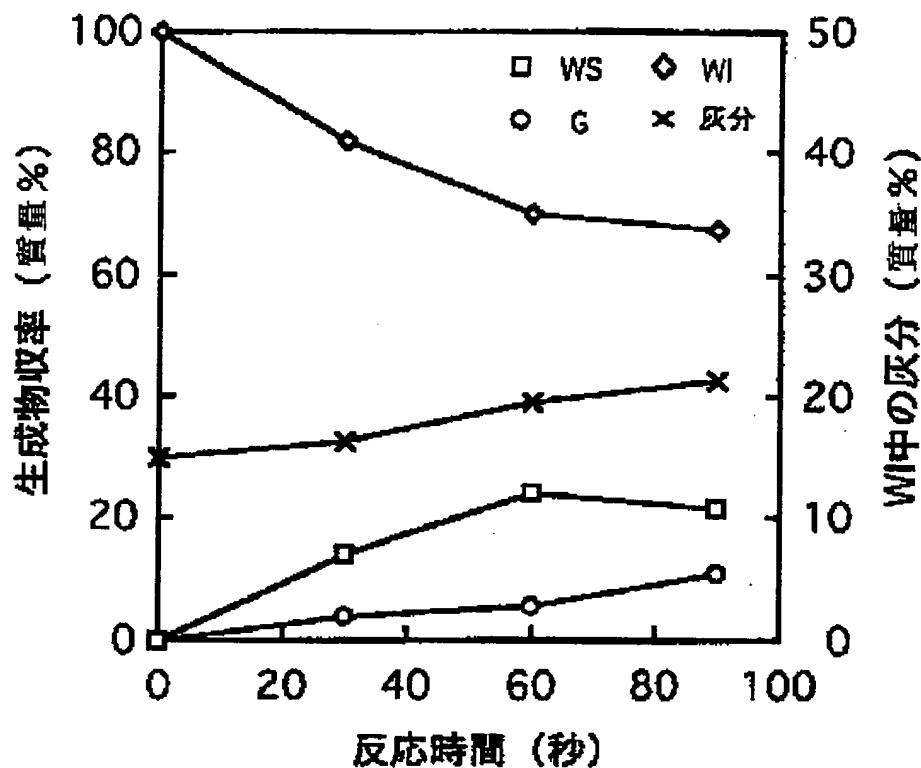
[Claim(s)]

[Claim 1] The manufacture approach of the silica raw material characterized by processing chaff powder for at least 30 seconds with 150-450-degree C pressurization hot water.

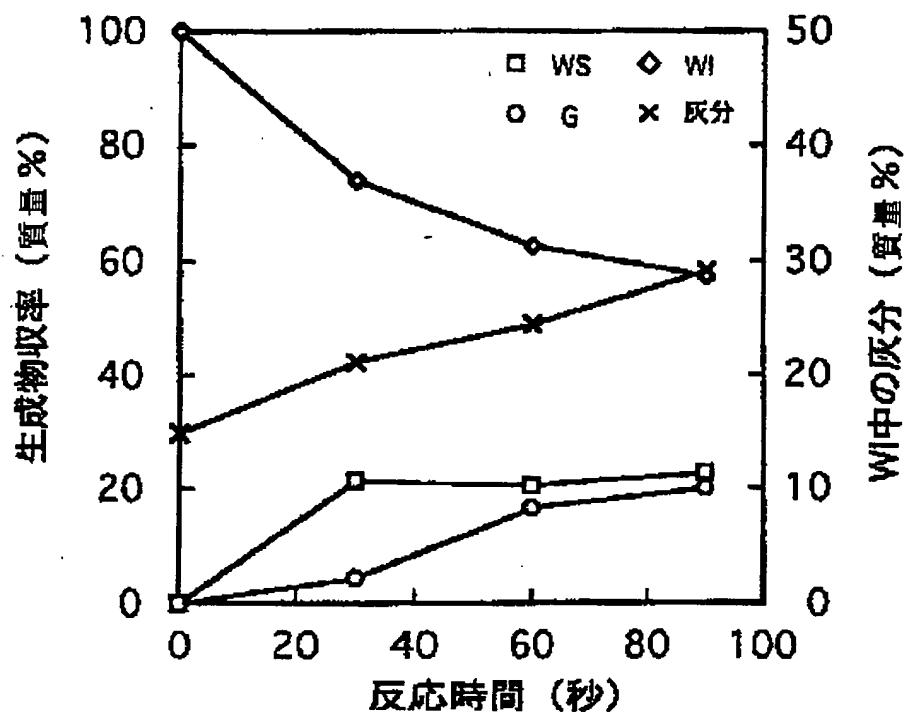
[Claim 2] The manufacture approach of a silica raw material according to claim 1 of adjusting the content rate of the silica component in the silica raw material generated by controlling reaction temperature and reaction time, and an organic part.

DRAWINGS

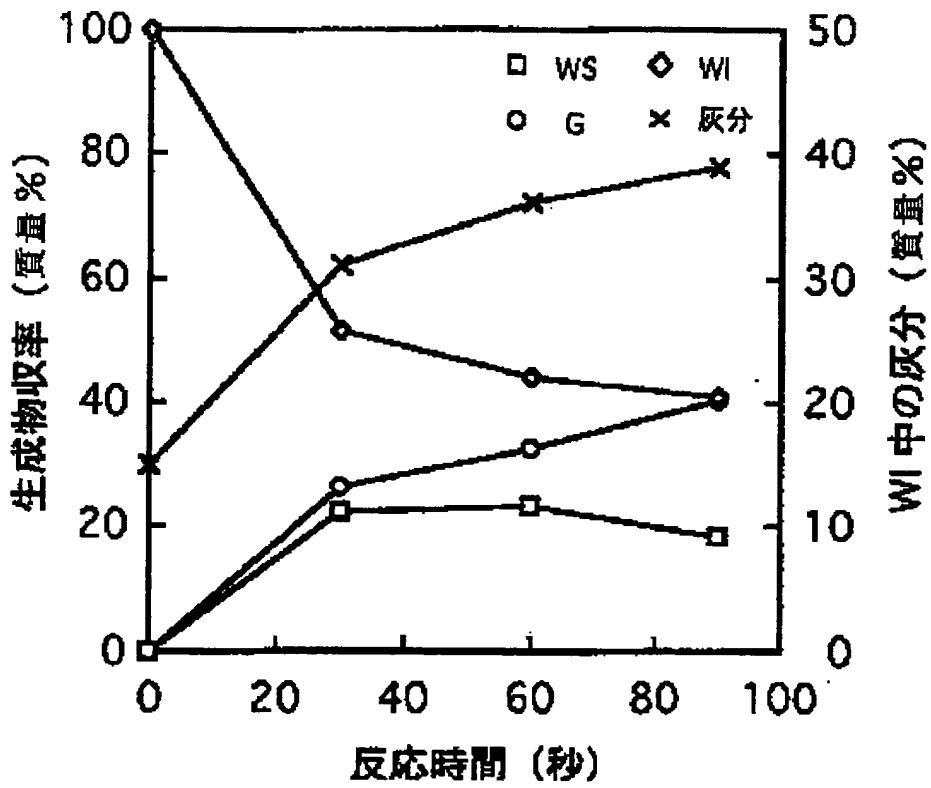
[Drawing 1]



[Drawing 2]



[Drawing 3]



[Drawing 4]

